Inductive Couple Plasma Atomic Emission Spectrometry (ICP-AES) for Pollution Monitoring

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Lecture – 16
Instrumentation for ICP AES-VIII-Instruments

We were discussing about the advantages of plasma source, and I had mentioned to you that high temperatures ensure complete atomization hence fewer interferences are found in ICP that is first advantage. So, one does not have to know quite a lot of chemistry. If one wants to determine any element in a in an ICP. So, long as the sample is in the liquid form which can be introduced directly into the I say plasma the you need not worry too much about the interference in occurring in ICP AAS.

This is not the case with spectrophotometry or atomic absorption you have to know a little bit more chemistry, but an operator can operate an ICP without much knowledge of chemistry also that is one of the main advantage. So secondly, atomization occurs in a chemically inert environment because it is argon is not is not it. So, long as we are using argon it is a linear element. So, there will not be many chemical reactions taking place inside the plasma.

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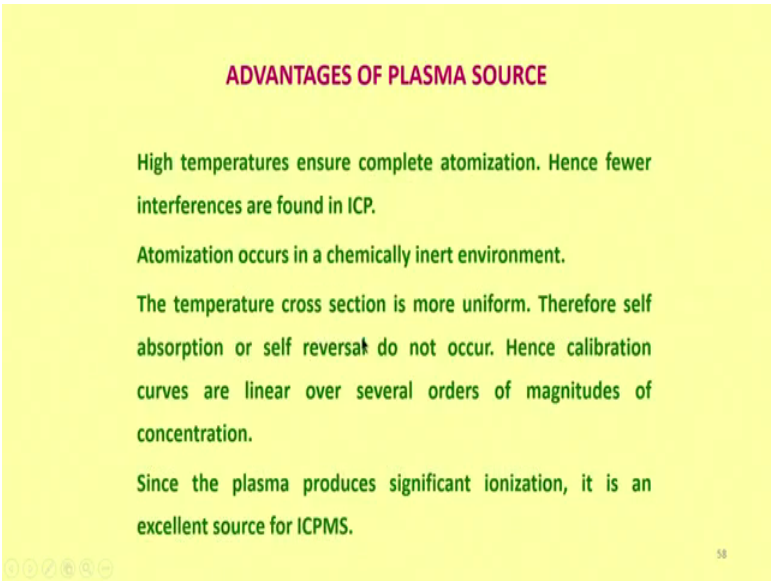

So, the third advantage is temperature cross section is more uniform. I had shown you this figure know.

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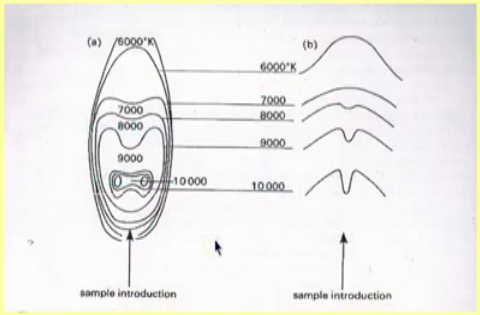

Figure ...(a)Approximate temperatures in degrees Kelvin in the ICP flame. (b)Cross-sectional view of temperature distribution within ICP flame at varying heights.

Here you can see the temperature cross section is quite uniform here, what does it mean? It means the efficiency of the plasma does not change much if you are off by 1 or 2 millimeter. Measuring window, you have a sort of capability to move a little bit this side that side without affecting the sensitivity. So, the calibration curves are also linear over several orders of magnitude unlike spectrophotometry or ICP. Since the plasma produces significant ionization it is an excellent source for ICPMS also why because the ionized atoms are easy to determine qualitatively also.

So, ICPMS is a sort of hyphenated technique, where inductive couple plasma is coupled with mass spectrometry for identification as well as quantification of the pollutants. So, this last sentence I have not taught you about mass spectrometry, but I we will be doing it some time maybe along with gc or hplc. So, excellent source for ICPMS is a lot of commercial instruments are already available in the market, known as the ICPMS instruments.

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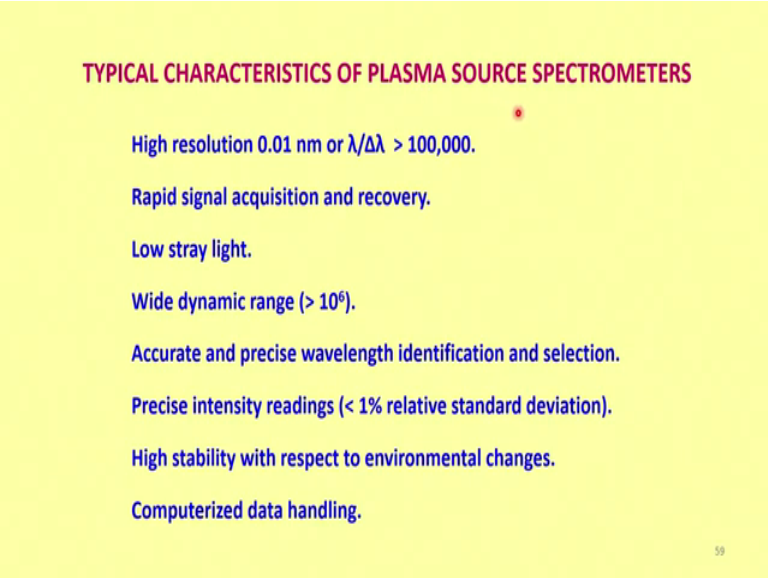

So, the typical characteristics of plasma include high resolution rapid signal acquisition, low stray light, wide dynamic range, accurate and precise wavelength precise intensity readings, high stability and computerized data handling. So, all these things combined together make the ICP one of the most versatile techniques for daily routine analysis or something like that.

So, there are as I had explained to you earlier there are various kinds of equipment's instruments and the one most suitable for a research laboratory or academic institution is a sequential ICP instruments. So, what does a sequential ICP instrument do it does one element at a time. So, only thing is you have to set the instrument for measuring only one element using one wavelength so one window. So, you do not need very high accuracy of the wavelength because it is will be only one wavelength, across the continuum of argon and you can use simple gratings using about 2400 or 3600 grooves per millimetre that is enough.

So, scanning how do you do the scanning? Then you set up the plasma start introducing the sample it will start giving you the signal, and you have to choose the wavelength from the computer and that is done by moving the grating. So, you can move the grating and then the dial on the instrument will show you what wavelength you are collecting. So, it will show you the wave length and from the database or the manuals from the

instrument manufacturer you have to choose the correct wavelength for each element you want to do for example, if you want to do copper go for 224.7 nanometer.

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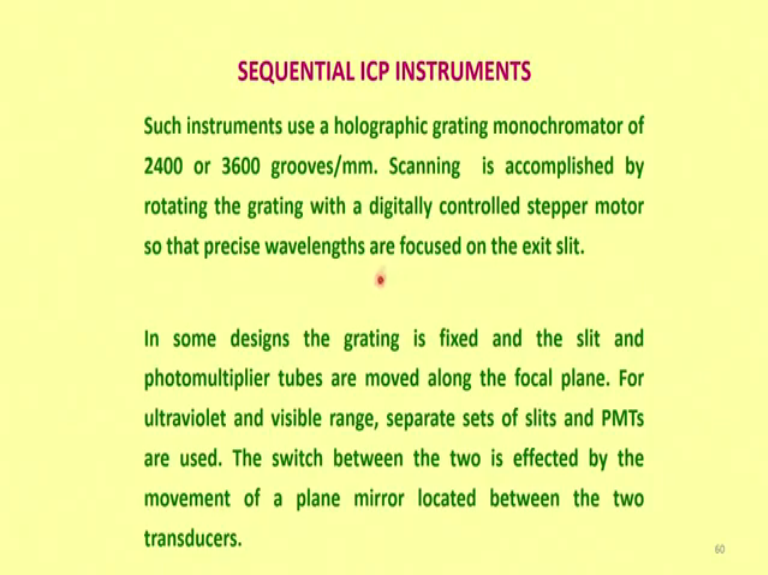

If it is calcium or sodium go for 589, like that we can adjust the wavelength for measurement before we continue with the analysis.

So, this rotating of the grating or in effect collecting a particular wavelength emission wavelength is controlled digitally using a stepper motor. So, a stepper motor runs on electricity and you can provide one millimetre by 1 milliamps, 2 milliamps, 5 millions, 10 milliamps. So, it will move the grating a little bit to arrive at the moment grating moves a little bit the dial and the wavelengths will show different wavelength. So, you keep on moving the grating using a stepper motor until you get the correct wavelength for your measurement.

So, the precise wavelengths are normally automatically focused on the exit slit that is Rowland circle. That we have already discussed number of times. So, in some designs the grating is fixed and the slit and photomultiplier tubes are moved along with the focal plane for ultraviolet visible range separate sets of sets slits and the photomultiplier tubes are used. So, the switch between the 2 that is PMTs is affected by the movement of the plane mirror located between the 2 transducers you can look up the manufacturers manual to understand what this means, but it is nothing very great rocket science.

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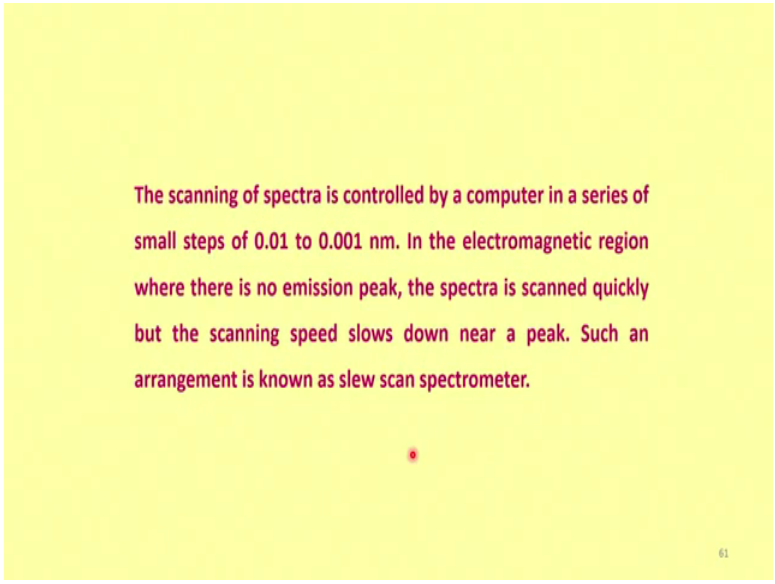

So, the scanning of the spectra is controlled by in small steps from 0.01 to 0.001 nanometer. So, normally for analytical purposes we need highly we do not need such high accuracy because optical window is transparent in the plasma as well as the wavelength plus or minus 01 to 001 that is 10 times order is not it. So, 10 times the order of slight inaccuracy does not matter as long as you are measurement wavelength is within that range. So, in the electromagnetic region where there is no emission peak suppose there is no emission peak. Then the what you should do is suppose you want to determine a particular element you do not know whether it is available or not in their sample, you want to determine cadmium in seawater or gold in seawater or thallium in urine. You do not know whether because everybody will not be having those components in their effluents or samples. So, if you do not know what element is there to determine first-hand what you should do you scan the wavelength see where you get the signal and match that sig wavelength with respect to the database available for which element in the emission line matches. So, pass and fail test is very simple straightaway you can go ahead and do it.

So, the spectra what we normally do we do an ICP for a known sample, we scan the whole wavelength spectrum. So, among them we suspect what are the elements present and then we choose the wavelengths for the quantitative determination. So, the spectra is scanned quickly first and the scanning speed I can reduce near the emission wavelength that is to pinpoint the exact wavelength where the measurements are to be made.

As I nearer as I go nearer the actual measurement for example, to 24.7 nanometer for copper so, as I go to 224 now I had to move I can move from one 80 to 220 very fast, and then to 220 to 224 nanometer I can move slowly and I can move still slowly between 224 and 224.7 nanometers. That is how the scanning in instruments are very useful because we can exactly pinpoint where is the emission wavelength occurring.

So, such an arrangement of the stepper motor is known as slew scan spectrometer, slew s l e w slew scan spectrometer. So, that is also part of these things so if you want to determine want to buy an instrument you better check whether if there is a slew scan spectrometer or not.

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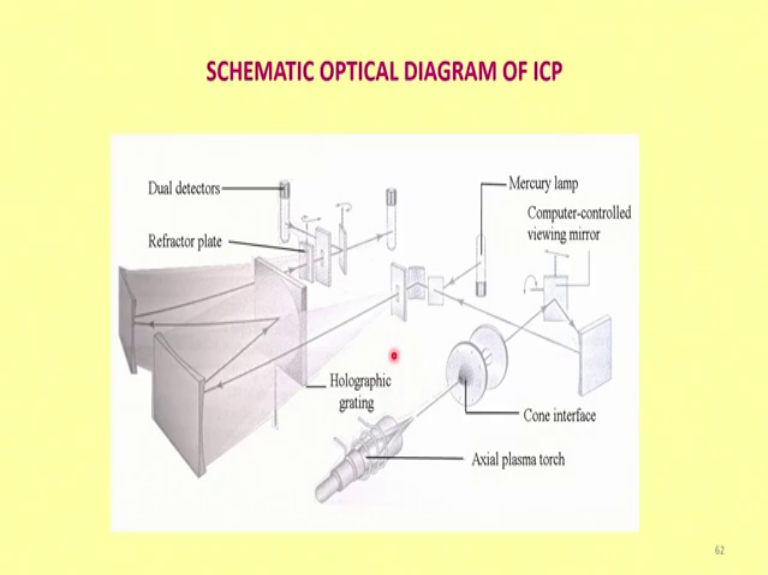

If it is not there you ask the manufacturer how do I go choose the correct wavelength etc they may offer you this slew scan spectrometer or a separate instrument for pinpointing the emission wavelength.

So, this is the schematic optical diagram of slew scan arrangement and you can see here excel plasma torches here and then we have the detectors, reactor plates, reflector plates, and then holographic grating is here this one you can see that it is almost flat you cannot really make out the grooves because the 2600 grooves in 1 centimetre or is something very rare. So, you do not our eyes are not sensitive enough to to see the grooves.

So, it looks like a mirror only, but it is a holographic grating and then what we have here is a mercury lamp; that means, the radiation from mercury lamp is passed along with our plasma; into the spectrometer optical arrangement. So, what it means actually is I have a sample here. So, the sample will be entering the plasma here and then it is exhale plasma torch cone interface is here, computer-controlled viewing mirror that is your wavelength setup and then this earth radiation comes through like this and it combines with a mercury lamp radiation.

So, mercury lamp radiation gives you specific wavelengths about 6 lines 220, 253 nanometer is one of the most used reference wavelength for all ICP, because what you need is a mercury lamp, every mercury lamp gives you about 5 or 6 lines I can in my previous course I have given you all those wavelengths and right now most of the instruments use mercury lamp 253.7 nanometer wavelength as a reference wavelength.

So, you have to adjust the emission with the mercury lamp to 253.7 if that fixes then all other wavelengths are automatically stand fixed. So, that is how the reference radiation mercury lamp is automatically included here so the sample radiation emission and then along with that it will be combined and it will go through these mirrors. This is Czerny turner instrument and grating and it goes to reflector plate dual detectors and then the photomultiplier tube, that is what it means and then everything is measured. So, this is the schematic optical diagram of ICP.

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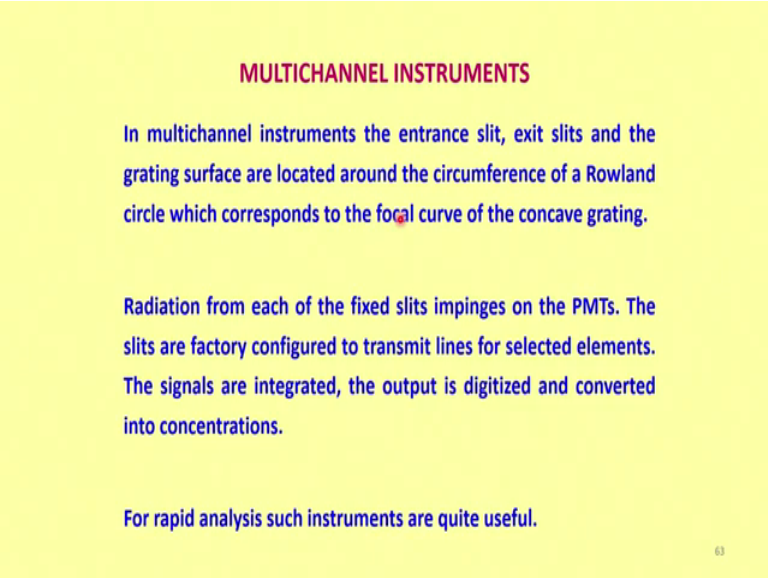

So, for multi-channel instruments what do we do? We want to measure in one sample several elements. So, what we usually look for is the concentrations of the elements in a given sample. The sample is repeated in resume you may assume that there is a foundry which produces special alloys and it does the same special alloy day in and day out day in and day out; that means, the components of the alloy remain the same. So, long as the industry is running.

So, the requirement of chemical analysis is also sort of fixed. So, in such cases we go for multi-channel instruments you tell them before buying the instrument you tell the manufacturer that I want to determine nickel, I want to determine chromium, I want to determine manganese, or I want to determine iron all in one sample. So, what they will do the manufacturer will cut fix the windows in such a way that mercury lamp, cadmium lamp, nickel lamp, nickel not lamps emission except mercury all other emission lines correspond to your requirement.

So, the point is you can do it so long as you have that multi-channel instrument, you do not have any freedom to check the unknown samples or some other element apart from what you have already decided to monitor. So, the all the detectors are also fixed to Rowland circle which corresponds to the focal curve of the concave grating. So, radiation from each of the fixed slit impinges on the PMTs. So, that is photomultiplier tubes the slips are factory configured you do not have to do anything and the to trial that is required to transmit lines for selected elements only

So, the signals are integrated and the output is digitized and that is converted into concentrations. So, for rapid analysis such instruments are quite useful for example, if you if you are in the laboratory the production man will just come bring you the sample and he just would not he may be just holding the sample in a furnace in a mol10 state he wants to cast into different products. So, the molten sample itself can be brought to the laboratory to determine all the multi-channel elements that you want to determine.

So, a complete multi-channel element analysis can be undertaken within 30 seconds. That is the beauty of ICPAAS, that is the up to 30 elements you can determine in one shot. So, the sample consumption will be hardly 0.5 ml of the sample 0.5 ml.

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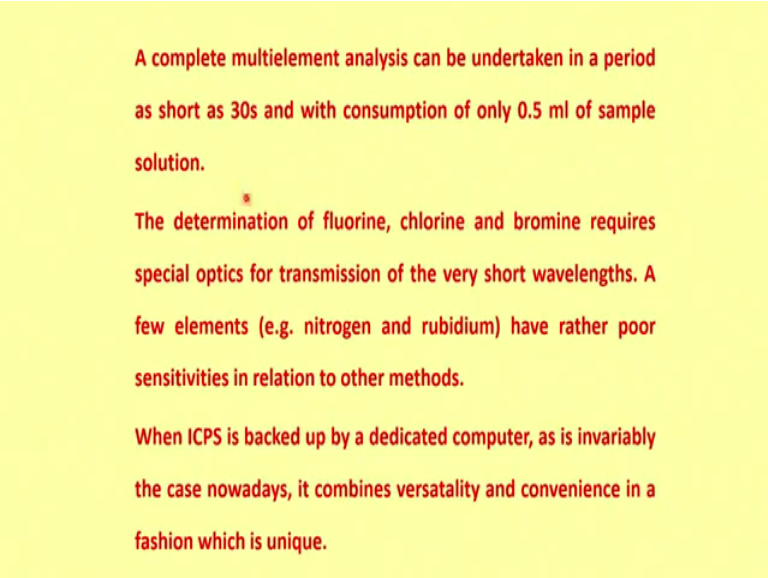

Not even 1 ml. So, their determination of these non-metallic elements such as fluorine, chlorine, bromine etcetera. They require special optics for the transmission of very short wavelengths normally, especially whenever you want to determine fluorine for example, chlorine, bromine and these things have got these elements have got characteristic emission lines in the ultraviolet rain or vacuum ultraviolet rain less than 180 nanometer. So, it requires special optics not even glass me quartz may not work, but some elements like nitrogen and rubidium they have rather poor sensitivity also you cannot determine with a very high accuracy and very low concentrations.

So, the ICP is not the panacea for all elemental analysis metals yes and metalloids yes take a look at the periodic table find out which are the metals and which are the metalloids that can be determined by ICP. And gaseous elements like fluorine, chlorine and bromine all these things are slightly difficult to analyze because they need special optics to isolate the emission wavelengths.

So, when ICPs is backed by a dedicated computer as is invariably the case 99 percent of the ICP instruments come with a microprocessor-controlled systems and it combines versatility and convenience in a fashion which is unique. Versatility is there convenience is there, and then both of them are combined to give you a beautiful experience of chemical analysis using metallic elements and non-metallic elements also metalloids and this is the multi-channel arrangement

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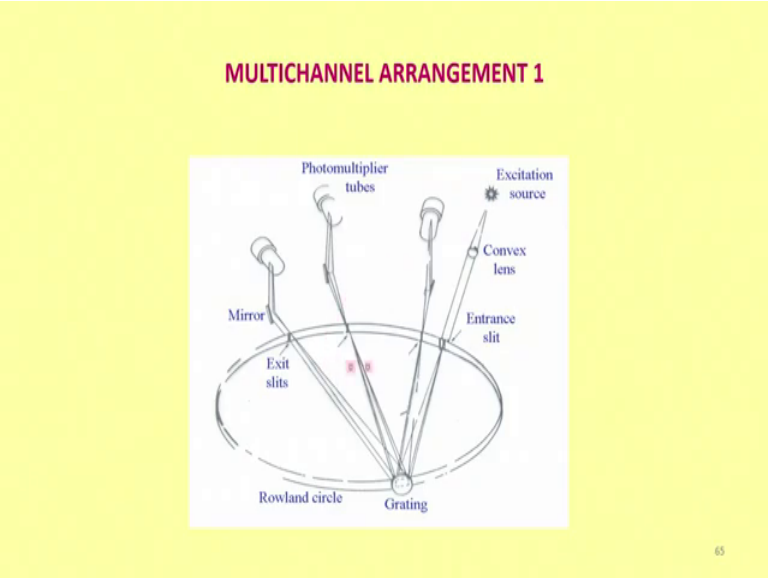

I had shown you once earlier while dealing with the optics here it is just the Rowland circle the grating is there. So, grating fixes the multi-channel wavelength one here, one here one, here one, here these are the slits and behind that there is a mirror in all these places there is a mirror to focus the radiation onto the in detector. So, these are this one, this one, in this one, they are all multi PMT photomultiplier tubes, which are the detectors for AAS this is a multi-channel arrangement that is one type.

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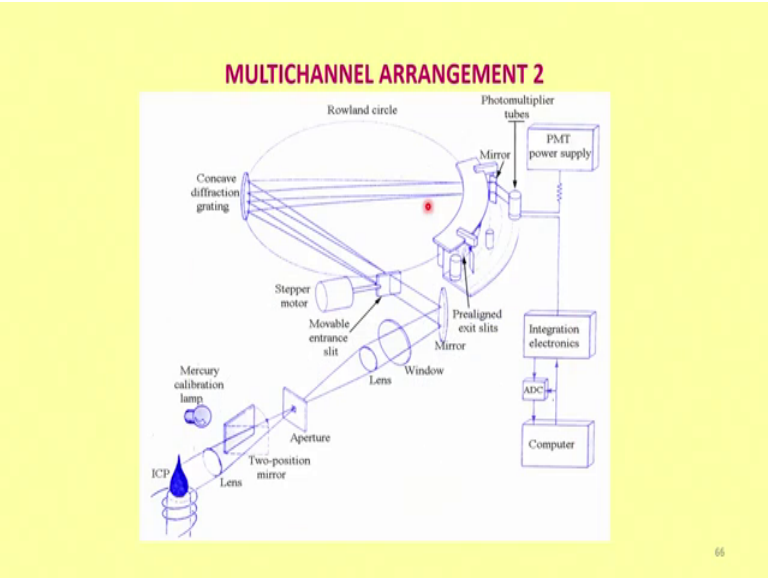

Another type is like this total system arrangement. Here I am showing you the PMT as well as integration electronics, analog to digital converter and computer also and all these things I have already explained to you earlier using optics and during the discussion on optics.

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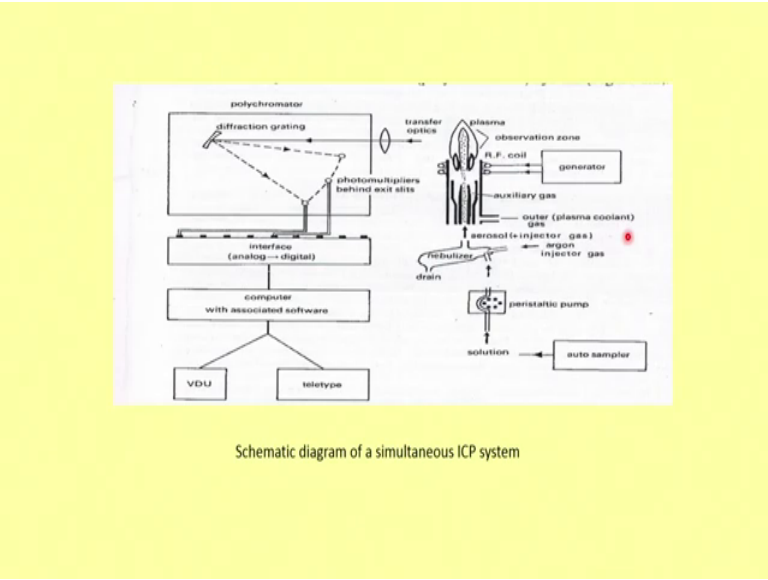

Schematic diagram of a simultaneous ICP system

So, this is another way of doing this multichannel instruments, simultaneous ICP here also the computer is here with associated software. All the software needs to be written inside the computer so that you can explore different options of mechanical as well as electronic controlling of the measurements. So, all other things remain the same for example, here is there is a diffraction grating, transfer optics and then photomultiplier tubes behind the exit slits. All these things are quite possible to use such instruments and this is only a schematic diagram, here I have a peristaltic pump also from the pump a tray reaches the Nebulizer and drain and may explain to you that the drain is always a requirement in both ICP and atomic absorption spectrometer.

So, so far what I have discussed with you is about the ICP. Now I have a feeling that what teaching you only this much does not really give you an insight of the operation of the ICP. So, I have included one more chapter on the applications, if you do not know the applications of a particular system you cannot use it efficiently that is as simple as that. So, we will proceed to the next module of our course that is applications of ICP AAS atomic emission spectrometry. So, we will continue our discussion right away.